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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE  
APPLICATION FOR LETTERS PATENT  
(UTILITY PATENT)

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INVENTION TITLE:

REJUVENATION OF REFRACTORY METAL  
PRODUCTS

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TO: BOX PATENT APPLICATION  
Commissioner for Patents  
Washington, D.C. 20231

Sir:

Your applicant(s), named above hereby petition(s) for  
grant of a utility patent to him(them) or any assignee(s) of  
record, at the time of issuance, for an invention more  
particularly described in the following specification and  
claims, with the accompanying drawings, verified by the  
accompanying Declaration and entitled:

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**TITLE****REJUVENATION OF REFRACTORY METAL PRODUCTS****CROSS REFERENCE TO RELATED APPLICATIONS**

- 1 This application claims priority of Provisional Application Number 60/268,742, entitled "REJUVENATION OF SPUTTERING TARGETS" filed on February 14, 2001, and which is incorporated herein by reference.

**FIELD AND BACKGROUND OF THE INVENTION**

2 The purpose of the invention is to decrease the recycling cost of refractory metal products, and in particular, rejuvenating sputtering targets having backing plate structures attached.

3 For example, sputtering targets of high temperature materials, such as tantalum and other refractory metals (Ta, Nb, Ti, Mo, Zr, metals and alloys; hydrides, nitrides and other compounds thereof) used in integrated circuit manufacture and other electrical, magnetic and optical product manufacture usually are eroded in a non-uniform way during the process of sputtering which leads to a race track like trench on the operating side of the target. In order to prevent any contamination of the substrates or catastrophic break-through of coolant fluids behind the target, the targets generally are withdrawn from service well before the refractory sputter metal is penetrated, accepting the need for a new target after only a minor portion of the sputter metal has been consumed. The major part of the sputter target can be resold only at scrap price or recycled with difficulty and apart from this, the backing plate of the target needs to be removed and may be re-bonded to a new sputter metal plate for recycling.

4 It is a principal object of the invention to replace such current recycling practice by rejuvenation of sputtering targets as described below.

5 It is an object of the invention to improve the cost and speed of getting used sputtering targets back into service.

6 It is a further object of the invention to establish a microstructure of the fill zone at least as good as on the balance of the target.

### SUMMARY OF THE INVENTION

7 The present invention is a method to rejuvenate surfaces of used refractory metal products by filling consumed surface areas with consolidated powder metal. For example, a race track trench or other erosion zone is produced on the face of a sputtering target after numerous non-uniform bombardments of argon atoms. The consumed surface is rejuvenated by the placement or deposition of sputter metal and sinter bonding by laser or EB heating for sintering or plasma discharge coupled with deposition. Use of these methods will yield a fully dense coating. This avoids the need for decoupling the tantalum from the copper, filling the erosion zone of the tantalum plate with tantalum powder and HIP (hot isostatic pressing) bonding and reassembly. In the case of laser or EB scan sintering or plasma discharge coupled with deposition the target can be rejuvenated without separating the backing plate from the target. The various forms of rejuvenation produce a filled erosion zone with microstructure similar to the balance of the target.

8 The invention can be applied to refractory metal products generally (whether or not mounted on a non-refractory metal carrier) that are subject to non-uniform erosion, etching, chipping or other metal loss. The form of

such refractory metal products can be as plate, rod, cylinder, block or other forms apart from sputter targets. The process can be applied to, for example, x-ray disks or targets (molybdenum plate on carbon backing).

- 9 The rejuvenation of a refractory metal product (e.g. tantalum target) eliminates the need to recycle the whole product after only a minor share of the product has been consumed. Such rejuvenation can be more economical than recycling the whole target. Separation of the bonded backing plate (e.g. copper), if any, may not be needed. This rejuvenation can be practiced repeatedly, as many times as desired.

- 10 Other objects, features and advantages will be apparent from the following detailed description of preferred embodiments taken in conjunction with the accompanying drawings in which:

#### BRIEF DESCRIPTION OF THE DRAWINGS

- 11 FIG. 1 shows a cross section of typical target and backing plate;
- 12 FIG. 2 shows a face view including a usual erosion zone;
- 13 FIG. 3 is a block diagram of the rejuvenation process; and
- 14 FIG. 4 shows in outline form a vacuum or inert gas chamber set-up for practice of the invention.

#### DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

- 15 Now turning to FIGS. 1 and 2, a tantalum (Ta) sputter plate 12 bonded to a copper (Cu) backing plate 14 is

presented to illustrate the rejuvenation process of the present invention. In addition to the backing plate, the sputter target may include additional complexity such as bonded-on water cooling coils 16 or even be part of a large cooling liquid reservoir and/or have complex flanges and mechanical and electrical attaching structures. 18 indicates a typical racetrack form erosion zone or consumed area on the target surface 20 of the sputter plate 12 arising from sputtering usage.

16 A flow chart of the implementation of the preferred embodiment of the present invention is illustrated in FIG. 3. A vacuum 22 or inert gas zone 24 is established for a used Ta-Cu target 26 assembly. The erosion zone 18 or consumed area of the sputter plate 12, as shown in FIG. 2, is filled with powders of the sputter metal. The powders are bonded or sintered 30 to the sputter plate 12 by laser or electron beam raster scanning to melt powder surfaces, but not complete particles or the entire particle that act as nuclei for grain growth. The melting can be done during powder deposition or after deposition on a layer-on-layer basis. A powder derived foil can also be pre-made and laid into the trench. In all cases the fill is sintered for self bonding and adhesion to the target and leveled off by machining, sanding or other abrasion etching and/or a burn-in sputtering process.

17 The following is one of several examples of how the invention can be implemented.

18 As shown in FIG. 4, a sputtering target 10 can be placed in a vacuum chamber 32 evacuated atmospheric pressure purified inert gas (argon) atmosphere utilizing conventional pump 34 and gas back-fill apparatus 36 with valve 38. A powder feeder 40 comprising multiple nozzles 42 can insert multiple high velocity streams of Ta powder of -100 to 325 mesh to the erosion zone 18 or consumed area. The powder feeder 40 can scan along the erosion zone 18 or the target

can be moved relative to a fixed powder feeder. A 15-20 KW (preferably 20-25) laser beam 44 formed by a laser 45 and conventional scan optics 46, 48 which can be wholly in the chamber 32 or partly outside the chamber 32 using a window for beam passage can be traced in raster scan fashion over the erosion zone 18, as the powder falls, to melt powder particle surfaces and enable particle to particle bonding and bonding to the base of the erosion zone continuously and repeatedly around the zone 18 until it is filled. Powder mass calculations and/or optical monitors can be used to determine completion and a cut-off of filling.

- 19 One form of equipment usable for such processing is the Lasform brand direct metal deposition system of AeroMet Corp., as described, e.g., in Abbott et al., "Laser Forming Titanium Components" in the May 1998 issue of Advanced Metals & Processes and Arcella et al., "Producing Titanium Aerospace Components From Powder Using Laser Forming," Journal of Metals (May 2000), pp. 28-30.

- 20 The laser can provide post-fill heating to complete the sintering. Separate target heaters can be used to preheat the target or provide additional heat during the rejuvenation.

- 21 The various forms of rejuvenation produce a filled erosion zone or consumed area with microstructure similar to the balance of the target. For example, filled erosion zone specimens from a sputtering target were analyzed for the electron beam raster scanning method. The hardness was typical for rolled and annealed tantalum plate with normal variation. The filled erosion zones were substantial free of porosity and inclusions. The yield strength and ultimate yield strength met ASTM requirements.

22 In another embodiment of the invention, the well known process of plasma deposition can be utilized to combine the powder placement and fusing steps.

23 It will now be apparent to those skilled in the art that other embodiments, improvements, details, and uses can be made consistent with the letter and spirit of the foregoing disclosure and within the scope of this patent, which is limited only by the following claims, construed in accordance with the patent law, including the doctrine of equivalents.

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